

## CLAIMS

What is claimed is:

1. A process for producing an ionic crosslinked fibrous material, the process comprising applying a polyelectrolyte to an ionic fibrous material to form an ionic crosslinked fibrous material, wherein the polyelectrolyte has a charge opposite that of the ionic fibrous material.
2. The process of Claim 1, wherein the polyelectrolyte comprises one of a polycation and a polyanion.
3. The process of Claim 2, wherein the polycation is formed by reacting a polymer with a cationizing agent.
4. The process of Claim 3, wherein the polymer comprises a polysaccharide.
5. The process of Claim 1, wherein the ionic fibrous material comprises an anionic fibrous material.
6. The process of Claim 5, wherein the anionic fibrous material is formed by reacting a fibrous material with a reactive anion.
7. The process of Claim 1, wherein the fibrous material is selected from the group consisting of a synthetic fibrous material and a natural fibrous material.
8. The process of Claim 7, wherein the natural fibrous material comprises a cellulosic fibrous material.
9. The process of Claim 8, wherein the cellulosic fibrous material comprises cotton.
10. The process of Claim 1, wherein the ionic crosslinked fibrous material exhibits an improved wrinkle recovery angle.
11. A process for producing an ionic crosslinked fibrous material, the process comprising:
  - (a) reacting a polymer with a cationizing agent to form a polycation;
  - (b) reacting a fibrous material with a reactive anion to form an anionic fibrous material; and
  - (c) applying the polycation to the anionic fibrous material to form an ionic crosslinked fibrous material.

12. The process of Claim 11, wherein the polycation is formed by:
- (a) mixing a polymer with a cationizing agent to form a reaction mixture;
  - (b) adding an aqueous alkaline solution to the reaction mixture to maintain the reaction mixture at a first pH range;
  - (c) stirring the reaction mixture for a period of time;
  - (d) heating the reaction mixture to a first temperature range for a period of time;
  - (e) cooling the reaction mixture to a second temperature range; and
  - (f) adding a protic acid to the reaction mixture to adjust the pH to a second pH range to form a polycation.
13. The process of Claim 12, wherein the polymer is selected from the group consisting of a deacetylated chitin and a partially deacetylated chitin.
14. The process of Claim 12, wherein the cationizing agent comprises a halogenated-hydroxyalkyl metal alkyl halide.
15. The process of Claim 14, wherein the halogenated-hydroxyalkyl metal alkyl halide is selected from the group consisting of 3-chloro-2-hydroxypropyl trimethyl ammonium chloride and epoxypropyl trimethyl ammonium chloride.
16. The process of Claim 12, wherein the aqueous alkaline solution comprises an aqueous alkali metal hydroxide solution.
17. The process of Claim 16, wherein the alkali metal hydroxide comprises sodium hydroxide.
18. The process of Claim 12, wherein the first pH range comprises a pH of about 10 to a pH of about 11.
19. The process of Claim 12, wherein the first temperature range comprises about 85°C to 105°C.
20. The process of Claim 12, wherein the second temperature range comprises about 20°C to about 25°C.
21. The process of Claim 12, wherein the second pH range comprises a pH of about 6.5 to a pH of about 7.5.

22. The process of Claim 12, wherein the protic acid comprises acetic acid.
23. The process of Claim 12, wherein the polycation comprises a cationized chitosan.
24. The process of Claim 11, wherein the anionic fibrous material is formed by:
- 5 (a) impregnating a fibrous material with an aqueous alkaline solution for a period of time at a first temperature range to form an alkali-treated fibrous material;
- (b) squeezing the alkali-treated fibrous material to a wet pickup of about 100%;
- 10 (c) drying the alkali-treated fibrous material at a second temperature range;
- (d) steeping the alkali-treated fibrous material in an aqueous solution of a reactive anion at a third temperature range for a period of time to form a treated fibrous material.
- 15 (e) squeezing the treated fibrous material to a wet pickup of about 100%;
- (f) sealing the treated fibrous material in a container; and
- (g) heating the sealed anionic fibrous material for a period of time to a fourth temperature range.
- 20 25. The process of Claim 24, wherein the aqueous alkaline solution of step (a) comprises an aqueous alkali metal hydroxide solution.
26. The process of claim 25, wherein the alkali metal hydroxide comprises sodium hydroxide.
- 25 27. The process of Claim 24, wherein the first temperature range comprises about 20°C to about 25°C.
28. The process of Claim 24, wherein the second temperature range comprises about 50°C to about 70°C.
29. The process of Claim 24, wherein the third temperature range comprises about 20°C to about 25°C.
- 30 30. The process of Claim 24, wherein the fourth temperature range comprises about 60°C to about 80°C.

31. The process of Claim 24, wherein the reactive anion of step (d) comprises a haloacetic acid.
32. The process of Claim 31, wherein the haloacetic acid is selected from the group consisting of chloroacetic acid and chlorosulfonic acid.
- 5 33. The process of Claim 24, further comprising the step of neutralizing the aqueous solution of the reactive anion of step (d) with a second alkaline solution.
34. The process of Claim 33, wherein a concentration of the second alkaline solution ranges from about 0 M to about 3.0 M.
- 10 35. The process of Claim 33, wherein the second alkaline solution comprises an alkali metal carbonate.
36. The process of Claim 35, wherein the alkali metal carbonate comprises sodium carbonate.
37. The process of Claim 24, wherein the process further comprises the steps of washing and drying the anionic fibrous material.
- 15 38. The process of Claim 24, wherein the anionic fibrous material comprises a carboxymethylated cellulosic material.
39. The process of claim 24, wherein the fibrous material comprises cotton.
40. The process of Claim 11, wherein the polycation is applied to the anionic fibrous material by:
- 20 (a) preparing an aqueous solution of the polycation;
- (b) padding the anionic fibrous material through the aqueous solution of the polycation to form a padded anionic fibrous material; and
- (c) drying the padded anionic fibrous material to form an ionic crosslinked fibrous material.
- 25 41. The process of Claim 40, wherein the aqueous solution of the polycation comprises an aqueous solution of cationized chitosan.
42. The process of Claim 40, wherein the concentration range comprises a weight percent concentration of the polycation of about 0% to about 6%.
- 30 43. The process of Claim 40, wherein the drying occurs at a temperature ranging from about 95°C to 115°C.

44. The process of Claim 40, wherein the anionic fibrous material comprises a carboxymethylated cellulosic material.
45. The process of Claim 40, further comprising padding the anionic fibrous material through an aqueous solution of the polycation to a wet pickup of about 100%.
46. The process of Claim 40, wherein the process is performed as a pad-dry process.
47. The process of Claim 40, wherein the fibrous material comprises cotton.
48. A process for producing an ionic crosslinked fibrous material, the process comprising:
- (a) mixing a cationizing agent with an alkaline compound to form a first reaction mixture;
  - (b) mixing the first reaction mixture with a reactive anion to form a second reaction mixture;
  - (c) padding a fibrous material through the second reaction mixture to form a treated fibrous material; and
  - (d) batching the treated fibrous material for a period of time at ambient temperature in a sealed container to form an ionic crosslinked fibrous material.
49. The process of Claim 48, wherein the cationizing agent comprises a halogenated-hydroxyalkyl metal alkyl halide.
50. The process of Claim 49, wherein the halogenated-hydroxyalkyl metal alkyl halide is selected from the group consisting of 3-chloro-2-hydroxypropyl trimethyl ammonium chloride and epoxypropyl trimethyl ammonium chloride.
51. The process of Claim 48, wherein the cationizing agent comprises a mixture of a halogenated-hydroxyalkyl metal alkyl halide and a reaction anion adduct, and wherein the halogenated-hydroxyalkyl metal alkyl halide is selected from the group consisting of 3-chloro-2-hydroxypropyl trimethyl ammonium chloride and epoxypropyl trimethyl ammonium chloride and the reaction anion is selected from the group consisting of chloroacetic acid and chlorosulfonic acid.

52. The process of Claim 48, wherein the alkaline compound comprises an alkali metal hydroxide.
53. The process of Claim 52, wherein the alkali metal hydroxide comprises sodium hydroxide.
- 5 54. The process of Claim 48, wherein the first reaction mixture comprises an epoxyalkyl metal alkyl halide.
55. The process of Claim 54, wherein the epoxyalkyl metal alkyl halide comprises epoxypropyl trimethyl ammonium chloride.
56. The process of Claim 48, wherein the reactive anion is selected from the group consisting of a haloacetic acid and an alkali metal haloalkyl sulfonate.
- 10 57. The process of Claim 56, wherein the haloacetic acid is selected from the group consisting of chloroacetic acid and chlorosulfonic acid.
58. The process of Claim 56, wherein the alkali metal haloalkyl sulfonate comprises sodium chloromethyl sulfonate.
- 15 59. The process of Claim 48, wherein the ambient temperature ranges from about 20°C to about 25°C.
60. The process of Claim 48, wherein a mol ratio range of the cationizing agent to the alkaline compound comprises about 1:2 to about 1:2.5.
- 20 61. The process of Claim 48, wherein the process is performed as a pad-batch process.
62. The process of Claim 48, wherein the fibrous material comprises cotton.
63. A process for producing an ionic crosslinked fibrous material, the process comprising:
- 25 (a) reacting a fibrous material with a reactive anion to form an anionic fibrous material;
- (b) mixing a cationizing agent with an alkaline compound to form a first reaction mixture;
- (c) padding the anionic fibrous material through the first reaction mixture to form a treated anionic fibrous material; and
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- (d) batching the treated anionic fibrous material for a period of time at ambient temperature in a sealed container to form an ionic crosslinked fibrous material.
- 5 64. The process of Claim 63, wherein the anionic fibrous material is formed by the process comprising:
  - (a) impregnating a fibrous material with an aqueous alkaline solution for a period of time at a first temperature range to form an alkali-treated fibrous material;
  - 10 (b) squeezing the alkali-treated fibrous material to a wet pickup of about 100%;
  - (c) drying the alkali-treated fibrous material at a second temperature range;
  - (d) steeping the alkali-treated fibrous material in an aqueous solution of a reactive anion at a third temperature range for a period of time to form a treated fibrous material.
  - 15 (e) squeezing the treated fibrous material to a wet pickup of about 100%;
  - (f) sealing the treated fibrous material in a container; and
  - 20 (g) heating the sealed anionic fibrous material for a period of time to a fourth temperature range.
- 65. The process of Claim 64, wherein the aqueous alkaline solution of step (a) comprises an aqueous alkali metal hydroxide solution.
- 66. The process of Claim 65, wherein the alkali metal hydroxide comprises sodium hydroxide.
- 25 67. The process of Claim 64, wherein the first temperature range comprises about 20°C to about 25°C.
- 68. The process of Claim 64, wherein the second temperature range comprises about 50°C to about 70°C.
- 30 69. The process of Claim 64, wherein the third temperature range comprises about 20°C to about 25°C.

70. The process of Claim 64, wherein the fourth temperature range comprises about 60°C to about 80°C.
- 5 71. The process of Claim 64, wherein the reactive anion is selected from the group consisting of a haloacetic acid and an alkali metal haloalkyl sulfonate.
72. The process of Claim 71, wherein the haloacetic acid comprises chloroacetic acid.
- 10 73. The process of Claim 71, wherein the alkali metal haloalkyl sulfonate comprises sodium chloromethyl sulfonate.
74. The process of Claim 64, further comprising the step of neutralizing the aqueous solution of the reactive anion of step (d) with a second alkaline compound.
- 15 75. The process of Claim 74, wherein a concentration of the second alkaline compound ranges from about 0 M to about 3.0 M.
76. The process of Claim 74, wherein the second alkaline compound comprises an alkali metal carbonate.
77. The process of Claim 76, wherein the alkali metal carbonate comprises sodium carbonate.
- 20 78. The process of Claim 64, wherein the process further comprises the steps of washing and drying the anionic fibrous material.
79. The process of Claim 64, wherein the anionic fibrous material comprises a carboxymethylated cellulosic material.
80. The process of claim 64, wherein the fibrous material comprises cotton.
- 25 81. The process of Claim 63, wherein the cationizing agent comprises a halogenated-hydroxyalkyl metal alkyl halide.
82. The process of Claim 81, wherein the halogenated-hydroxyalkyl metal alkyl halide comprises 3-chloro-2-hydroxypropyl trimethyl ammonium chloride.
- 30 83. The process of Claim 63, wherein the cationizing agent comprises a mixture of a halogenated-hydroxyalkyl metal alkyl halide and a reaction anion adduct, and wherein the halogenated-hydroxyalkyl metal alkyl



halide is selected from the group consisting of 3-chloro-2-hydroxypropyl trimethyl ammonium chloride and epoxypentyl trimethyl ammonium chloride, and the reactive anion is selected from the group consisting of chloroacetic acid and chlorosulfonic acid.

- 5     84.     The process of Claim 63, wherein the alkaline compound comprises an alkali metal hydroxide.
85.     The process of Claim 84, wherein the alkali metal hydroxide comprises sodium hydroxide.
86.     The process of Claim 63, wherein the first reaction mixture comprises an epoxyalkyl metal alkyl halide.
- 10     87.     The process of Claim 86, wherein the epoxyalkyl metal alkyl halide comprises epoxypentyl trimethyl ammonium chloride.
88.     The process of Claim 63, wherein a mol ratio range of the cationizing agent to the alkaline compound comprises a mol ratio of about 1:2 to a mol ratio of about 1:2.5.
- 15     89.     The process of Claim 63, wherein the process is performed as a pad-batch process.
90.     An ionic crosslinked fibrous material formed by the process of Claim 1.
91.     The ionic crosslinked fibrous material of Claim 90, wherein the ionic crosslinked fibrous material exhibits an improved wrinkle recovery angle.
- 20     92.     The ionic crosslinked fibrous material of Claim 90, wherein the ionic crosslinked fibrous material exhibits an improved wrinkle recovery angle without a loss of strength.
93.     A polycation formed by the process of Claim 12.
- 25     94.     The polycation of Claim 94, wherein the polycation comprises a cationized chitosan.
95.     The polycation of Claim 94, wherein the cationized chitosan exhibits cationization at the C<sub>6</sub> and ring hydroxyl sites.
96.     The polycation of Claim 94, wherein the reactivity of the ring amino group sites is preserved.
- 30     97.     A process for producing a cationized fibrous material, the process comprising:

- (a) preparing a first reaction mixture, wherein the first reaction mixture comprises a cationizing agent, an alkaline compound, and mixtures thereof;
- 5 (b) padding the fibrous material through the first reaction mixture to a wet pickup of about 100% to form a first padded fibrous material;
- (c) preparing a second reaction mixture, wherein the first reaction mixture comprises a cationizing agent, an alkaline compound, and mixtures thereof;
- 10 (d) padding the fibrous material through the second reaction mixture to a wet pickup of about 100% to form a second padded fibrous material; and
- (e) batching the padded fibrous material in a sealed container at a temperature range for a period of time to form a cationized fibrous material.
- 15 98. The process of Claim 97, wherein the cationizing agent comprises a halogenated-hydroxyalkyl alkylmetal halide.
99. The process of Claim 98, wherein the halogenated-hydroxyalkyl alkylmetal halide comprises 3-chloro-2-hydroxypropyl trimethyl ammonium chloride.
- 20 100. The process of Claim 97, wherein the alkaline compound comprises an alkali metal hydroxide.
101. The process of Claim 100, wherein the alkali metal hydroxide comprises sodium hydroxide.
102. The process of Claim 97, wherein the first reaction mixture comprises the cationizing agent only.
- 25 103. The process of Claim 97, wherein the first reaction mixture comprises the alkaline compound only.
104. The process of Claim 97, wherein the first reaction mixture further comprises an epoxyalkyl metal alkyl halide.
- 30 105. The process of Claim 104, wherein the epoxyalkyl metal alkyl halide comprises epoxypropyl trimethyl ammonium chloride.

106. The process of Claim 97, wherein the second reaction mixture comprises the cationizing agent only.
107. The process of Claim 97, wherein the second reaction mixture  
5 comprises the alkaline compound only.
108. The process of Claim 97, wherein the second reaction mixture further comprises an epoxyalkyl metal alkyl halide.
109. The process of Claim 108, wherein the epoxyalkyl metal alkyl halide comprises epoxypropyl trimethyl ammonium chloride.
- 10 110. The process of Claim 97, further comprising the steps of (a), (b), and (e) only.
111. The process of Claim 97, further comprising the step of drying the fibrous material after step (b).
112. The process of Claim 97, further comprising the step of adding an  
15 additive to the first reaction mixture, wherein the additive is selected from the group consisting of sodium lauryl sulfate, triethanol amine, ethylenediamine tetraacetic acid, butane tetracarboxylic acid, sodium thiosulfate, sodium tetraborate, sodium chloride, guanidine, diethylamine, and epichlorohydrin.
- 20 113. The process of Claim 97, further comprising the step of subjecting the fibrous material to a pretreating process prior to padding the fibrous material through the first reaction mixture, wherein the pretreating process comprises:
- 25 (a) soaking the fibrous material in a pretreatment solution at a temperature range for a period of time to form a pretreated fibrous material; and
- (b) removing the pretreatment solution from the pretreated fibrous material by one of:
- 30 (i) washing the pretreated fibrous material with a washing solution; and
- (ii) drying the pretreated fibrous material at a second temperature range.

114. The process of Claim 113, wherein the pretreatment solution is selected from the group consisting of guanidine, sodium hydroxide, potassium hydroxide, trimethylammonium hydroxide, aqueous ammonia, and liquid ammonia.
- 5 115. The process of Claim 113, wherein the first temperature range comprises about 20°C to about 25°C, under the proviso that the pretreatment solution does not comprise liquid ammonia.
116. The process of Claim 113, wherein the first temperature range comprises about -75°C to about -80°C, under the proviso that the pretreatment solution comprises liquid ammonia.
- 10 117. The process of Claim 113, wherein the washing solution is selected from the group consisting of water and guanidine.
118. The process of Claim 113, wherein the second temperature range comprises about 20°C to about 25°C.
- 15 119. The process of Claim 97, wherein the process is performed as a pad-batch process.
120. The process of Claim 97, wherein the fibrous material comprises cotton.
121. A process for producing a cationized fibrous material, the process comprising:
- 20 (a) mixing a cationizing agent and an alkaline compound to form a first reaction mixture;
- (b) waiting for a first period of time; and
- (c) adding a fibrous material to the first reaction mixture for a second period of time.
- 25 122. The process of Claim 121, wherein the cationizing agent comprises a halogenated-hydroxyalkyl metal alkyl halide.
123. The process of Claim 122, wherein the halogenated-hydroxyalkyl metal alkyl halide comprises 3-chloro-2-hydroxypropyl trimethyl ammonium chloride.
- 30 124. The process of Claim 121, wherein the alkaline compound comprises an alkali metal hydroxide.

125. The process of Claim 124, wherein the alkali metal hydroxide comprises sodium hydroxide.
126. The process of Claim 121, wherein the first period of time comprises  
5 from about 1 minute to about 15 minutes.
127. The process of Claim 121, wherein the second period of time comprises from about 80 minutes to about 100 minutes.
128. The process of Claim 121, further comprising the step of adding a second alkaline compound to the reaction mixture of step (c).
- 10 129. The process of Claim 128, wherein the second alkaline compound comprises an alkali metal carbonate.
130. The process of Claim 129, wherein the alkali metal carbonate comprises sodium carbonate.
131. The process of Claim 121, further comprising the step of adding an  
15 additive to the first reaction mixture, wherein the additive is selected from the group consisting of a NaOH/Na<sub>2</sub>CO<sub>3</sub> pH 12 buffer solution, triethanol amine, sodium chloride, sodium lauryl sulfate, ethylenediamine tetraacetic acid, and epichlorohydrin.
132. The process of Claim 121, further comprising the step of adding a  
20 solvent to the first reaction mixture, wherein the solvent is selected from the group consisting of acetone, methanol, ethanol, and isopropanol.
133. The process of Claim 121, further comprising the sequence of adding the fibrous material to the cationizing agent and then adding the alkaline compound.
- 25 134. The process of Claim 121, further comprising the sequence of adding the fibrous material to the alkaline compound and then adding the cationizing agent.
135. The process of Claim 121, wherein the process is performed as an exhaust fixation process.
- 30 136. The process of Claim 121, wherein the fibrous material comprises cotton.

137. A process for producing a cationized fibrous material, the process comprising:
- 5 (a) mixing a cationizing agent and an alkaline compound to form a first reaction mixture;
- (b) padding a fibrous material through the first reaction mixture to form a padded fibrous material;
- (c) drying the padded fibrous material at a first temperature range; and
- 10 (d) exposing the padded fibrous material to saturated steam at a second temperature range for a period of time.
138. The process of Claim 137, wherein the cationizing agent comprises a halogenated-hydroxyalkyl metal alkyl halide.
139. The process of Claim 138, wherein the halogenated-hydroxyalkyl metal alkyl halide comprises 3-chloro-2-hydroxypropyl trimethyl ammonium chloride.
- 15 140. The process of Claim 137, wherein the alkaline compound comprises an alkali metal hydroxide.
141. The process of Claim 140, wherein the alkali metal hydroxide comprises sodium hydroxide.
- 20 142. The process of Claim 137, wherein the first temperature range comprises about 35°C to about 45°C.
143. The process of Claim 137, wherein the second temperature range comprises about 95°C to about 105°C.
- 25 144. The process of Claim 137, further comprising the steps of (a), (b), and (d) only.
145. The process of Claim 137, wherein the process is performed as a pad-steam process.
146. The process of Claim 137, wherein the fibrous material comprises cotton.
- 30 147. A process for producing cationized fibrous material, the process comprising:

- (a) mixing a cationizing agent and an alkaline compound to form a first reaction mixture;
  - (b) padding a fibrous material through the first reaction mixture to a wet pickup of about 100% to form a padded fibrous material;
  - 5 (c) drying the padded fibrous material at a first temperature range for a first period of time; and
  - (d) curing the padded fibrous material at a second temperature range for a second period of time.
148. The process of Claim 147, wherein the cationizing agent comprises a  
10 halogenated-hydroxyalkyl metal alkyl halide.
149. The process of Claim 148, wherein the halogenated-hydroxyalkyl metal alkyl halide comprises 3-chloro-2-hydroxypropyl trimethyl ammonium chloride.
150. The process of Claim 147, wherein the alkaline compound comprises an  
15 alkali metal hydroxide.
151. The process of Claim 150, wherein the alkali metal hydroxide comprises sodium hydroxide.
152. The process of Claim 147, wherein a mol ratio range of the alkaline compound to the cationizing agent comprises about 0.50:1 to about  
20 2.5:1.
153. The process of Claim 147, further comprising adding an additive to the first reaction mixture, wherein the additive is selected from the group consisting of sodium chloride, sodium acetate, triethanol amine, and sodium lauryl sulfate.
- 25 154. The process of Claim 147, wherein the first temperature range comprises about 20°C to about 100°C.
155. The process of Claim 147, wherein the second temperature range comprises about 40°C to about 130°C.
156. The process of Claim 147, wherein the first period of time comprises  
30 about 1 minute to about 15 minutes.
157. The process of Claim 147, wherein the second period of time comprises about 1 minute to about 30 minutes.

158. The process of Claim 147, wherein the process is performed as a pad-dry-cure process.
159. The process of Claim 147, wherein the fibrous material comprises cotton.